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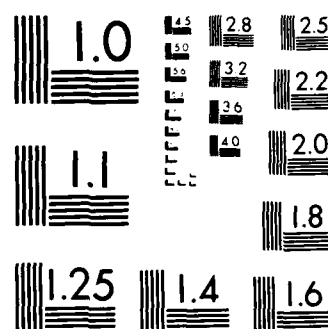
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PRECISE MEASUREMENT OF REFRACTIVE INDEX AND  
ABSORPTION COEFFICIENT OF NEAR MILLIMETER WAVE  
AND FAR INFRARED MATERIALS

FINAL REPORT

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MOHAMMED NURUL AFSAR AND KENNETH J. BUTTON

JUNE 1987

U. S. ARMY RESEARCH OFFICE  
RESEARCH TRIANGLE PARK, NORTH CAROLINA

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A quasi-optical technique, namely, dispersive Fourier transform spectrometry, has now been improved to provide high-precision continuous data of complex refractive index, complex dielectric permittivity and loss tangent of materials and complex magnetic permeability of ferrite materials at millimeter and submillimeter wavelengths. The use of a polarizing two beam interferometric technique allowed a broad frequency coverage in particularly between 6mm and 0.5mm range. Non-polar polymers such as polyethylene and teflon exhibit extremely low-loss characteristics in the entire millimeter wavelength range. Polar polymers such		

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as plexiglass and Nylon exhibit atleast 20 to 30 times higher loss compared to non-polar polymers. A massive birefringent effect ( $\Delta\epsilon' = 2.193$ ) is now observed for crystalline sapphire, which is atleast 10 times higher than crystalline quartz ( $\Delta\epsilon' = 0.2$ ). The resonance peak for ferrite materials move with applied magnetic field increments. Complex magnetic permeability values then can be seperated out completely experimentally. On hexagonal ferrites, the natural ferromagnetic resonance lies in the 1 mm range. A batch of specimens have been studied over the frequency range 60-450GHz under the sample exchange program organized by U.S. Army Harry Diamond Laboratories.

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We have extended a broad-band interferometric technique well into the 5mm range[1]. Various measures such as larger throughput, use of sensitive detectors, mechanical and electronic stability, and minimization of power losses enabled us to produce data of complex refractive index, complex dielectric permittivity, and loss tangent over the wavelength range 6 - 0.5mm. The dispersive Fourier transform spectroscopic technique developed by one of us (Afsar) [1-6] measures the phase as well as the amplitude, the analysis of which provides the absorption coefficient, the refractive index, the real and imaginary part of the dielectric permittivity and the loss tangent.

The millimeter-wave absorption in materials is dominated by the tail of the lattice vibration absorptions in the submillimeter and infrared region. Additionally, there are also Debye-type liquid lattice absorption present in the millimeter-wave regions. Commonly used non-polar polymers are polyethylene, polypropylene, polytetrafluorethylene (Teflon) and poly-4 methyl pentene-1. Because of their low-loss characteristics, they are already in use as windows and lenses in submillimeter wave and infrared spectrometers. Polypropylene came as a substitute for polyethylene in radio frequency region as well as in the submillimeter wavelength region. It is no longer necessary to mention the suitability of polyethylene in particular in the radio frequency and in the microwave frequency region except that the material is relatively softer compared to polypropylene. Both were used in a rather parallel fashion around 1-GHz region. Around the middle of the submillimeter waveband (2100 GHz,  $70 \text{ cm}^{-1}$ ), polyethylene has a very sharp intense absorption band, which forbids its use in the region 1,800 - 2,400 GHz ( $60\text{-}80 \text{ cm}^{-1}$ ). Teflon is inert to all organic liquids and known to possess very low-loss characteristics in the radio frequency region. The microwave loss-tangent value of unsintered polytetrafluorethylene is of the order of 40 micro-radians. At the mid-infrared region (6,000 GHz), a strong lattice absorption band precludes its use beyond 4,500 GHz ( $150 \text{ cm}^{-1}$ ). The mechanical property of teflon is somewhat poorer than polypropylene. The poly-4 methyl pentene-1 (trade name TPX as of Imperial Chemical Industries, England) is a clear glass like plastic and makes the material ideally suited for window applications in spectrometers. Once the instrument is equipped with TPX, it is very easy to make optical alignments. The refractive index of TPX in the visible frequency region (1.43) matches very very closely with its value in the submillimeter wavelength region (1.46). The material is much harder than three other non-polar polymers mentioned above. Moreover the TPX is resistant to deformation by heat.

The absorption data of polypropylene vary markedly. Commercially available polypropylene contains far more impurities than does polyethylene. There are not only such things as anti-oxidants and plasticizers which are normally added to improve the mechanical and electrical properties of the material, but also inadvertent impurities, such as catalyst residues. Some of these impurities are ionic which give rise to absorption beyond 3,000 GHz ( $100 \text{ cm}^{-1}$ ), and for this reason many commercially available polypropylene specimens are quite opaque in the submillimeter and mid-infrared region. One can say that in the strict sense there is no pure polymer and they all show wide ranges of molecular weight. Semicrystalline polymeric materials are made of crystalline regions spread throughout an amorphous matrix and are quite homogenous. The absorption coefficient of all polymers can also vary markedly with the composition, history and the temperature of the specimen. In the millimeter and submillimeter region, absorption processes in polymers arise from low-frequency intra molecular modes, inter molecular modes or lattice modes, amorphous region processes, relaxation and non-resonant processes, and from impurities accidentally or deliberately present in the material. The vibrations of highly localized groups of atoms contribute to the mid- and near-infrared absorption processes. In the submillimeter wave region, absorption due to chain twisting motions are also common for a long-chain molecule.

The infrared spectra of typical hydrocarbon polymers gives only second order differences between the spectra of very different materials, because the frequency of a CH- stretching mode are mainly determined by the nearly invariant bond force constant. This lead to a small frequency splitting or small variations in the spectral intensity. The infrared method is less sensitive to distant effects arising from the polymeric nature of the molecule. In the millimeter and near millimeter frequencies, spectral response for common polymers represents a monotonic function. All four non-polar polymers mentioned above exhibit extremely low-loss characteristics in the entire millimeter wavelength range. The trend of monotonic increase in absorption with increasing frequency is due to the amorphous continuum. The high density polyethylene exhibits the lowest absorptions. This material can now be classified as the lowest loss material in the millimeter wavelength range. It was not surprising to see the absorption data of our polypropylene to be of higher value. Both Teflon and TPX exhibit lower loss at 70GHz compared to polypropylene. Around 130 GHz and 200GHz their absorption become higher than that of polypropylene. One may try to assign the millimeter-wave absorptions of non-polar polymers are made up of contributions from tail of crystalline region contributions and amorphous region contributions. In non-polar polymers, the crystallinity depends on the nature of the specimen material and on the treatment given to it during forming. It is quite clear that the monotonic trend of increase in absorption with increasing frequency must be largely associated with amorphous regions. Teflon shows the lowest refractive index value( 1.439) among these four non-polar polymers. It has an intense and sharp absorption band centering around 6000 GHz, which precludes its use in the submillimeter wavelength range. The refraction spectrum falls to a value of 1.38 and then shows an anomalous dispersion through 6,000 GHz band.

For these low-loss non-polar polymers, the real part of the dielectric permittivity values are essentially square of their refractive index values. The loss tangent values for polyethylene lies in the range 360-440 micro-radians between 60 and 300 GHz.

Both Plexiglas and Acrylic are made with acrylic acid. It was not surprising to see both exhibiting similar absorption characteristics atleast 30 times higher than polyethylene. The plexiglass is a polymethyl methacrylate and the acrylic is a polymethacrylate. The polyamide( nylon) is made with adipic acid and hexamethylene-diamine. The nylon showed 20% more absorption values compared to plexiglass and acrylic. For these three polar polymers the absorption coefficient values rise monotonically and almost linearly in the 60-300 GHz region. These polar polymers are certainly lossy materials. Their strong absorptions are analogous to broad absorptions which were found in the 300-3000 GHz region for polar liquids. Nylon has a very high thermal stability, and is often used as a thermal insulator in many high- and low- temperature applications. None of these three polar polymers are suitable for window and lens applications because of their poor transmission( high absorption). It was not surprising to see nearly identical refraction spectra for both plexiglass and acrylic. The average value lies around 1.611. The refraction spectrum of nylon has a similar trend as methacrylates but values that are much higher. The real part of the dielectric permittivity values for these absorbing polar polymers are no longer square of their refraction values. The high absorption coefficient values significantly contributed to the real part of the permittivity values. The lowest loss tangent value is almost equal to 8100 micro-radians( for plexiglas and acrylic at 105GHz).

Ceramics such as Beryllium Oxide( $\text{BeO}$ ), Aluminium Oxide( $\text{Al}_2\text{O}_3$ ), Boron Nitride and Silicon Carbide are potential candidates as window materials in high temperature applications for high power millimeter wave sources such as gyrotrons and substrates in microprocessor industry. The lower absorption loss & high value of refractive index are vital in such applications in addition to the withstand

capability of heat. The high refractive index value and low absorption coefficient numbers make these ceramic materials to be additionally attractive for applications such as dielectric waveguides. In the past we have noticed that most ceramics exhibit a continuum type of monotonic increase in absorption with increase in frequency in the millimeter-wave regions in addition to the effect of the tail of the submillimeter wave and infrared lattice vibration absorptions. A crystalline material do not exhibit this amorphous continuum type of absorptions as does the polycrystalline cold- and hot-pressed sintered ceramics. But crystalline materials easily shatter or break in high temperature window applications and additionally they are bire-fringent. Our new study reveals intimate details, on relationship of degree of crystallinity of ceramics and the absorption loss characteristics. The absorption loss for a single crystal sapphire ( $\text{Al}_2\text{O}_3$ ) at millimeter wavelength region is very low. There is a small variation in the loss between the ordinary and the extra-ordinary ray data for an a-cut crystal. Again like many other millimeter wave material, the absorption values increases monotonically from 0.01 Neper/cm at 50 GHz to 0.15 Neper/cm at 300 GHz. Upto 250GHz the difference in absorption values is very small, then spectra cross over and the difference increase. A comparison of absorption data with polycrystalline alumina from AMPEX Corporation reveals that the crystallinity has reduced the absorption values to about a factor of three in the entire millimeter wave region. It might be wise to introduce some degree of crystallinity by hot pressing while sintering to reduce the absorption loss in polycrystalline alumina. The origin of this millimeter wave monotonic increase in absorption with increase in frequency in the single crystal sapphire is mainly due to the "tail" effect of the submillimeter wave and infrared lattice vibration bands. The comparison of ordinary ray and extra-ordinary ray refractive index for the single crystal sapphire shows a very strong and frequency dependent bire-fringent ( $\Delta n$ ) effect in the millimeter wave region. Initially refractive index values fall very sharply in both cases, but does not change much in the range 120-400GHz. Between 120-400 GHz the nature of spectra exhibit opposite trend, correlating to crossover point in their absorption spectra. However, the average difference in refractive index values is very large between the ordinary ray and the extra-ordinary ray spectra ( $\Delta n \approx 0.34$ ) and is at least seven times higher compared to single crystal quartz. The variation in the  $\Delta n$  values with increasing frequency is very interesting, since it shows a minimum and a maximum in the millimeter wavelength range. This also demonstrates the powerfullness of the dispersive Fourier transform spectroscopy. We can measure refractive index to one part in  $10^5$ . The variation of  $\Delta n$  can be seen, only because of the high resolution of the refractive index data. This high birefringent effect is a significant advantage in polarimeter applications, in well controlled beam rotating, beam modulating and phase shifting purposes. However this precludes the use of sapphire as a window and lens material. One could easily end up including significant error in measurements unless the orientation of the single crystal material is carefully selected. Again we observed the real part of the dielectric permittivity values to be square of their refractive index values for this low loss material. The  $\Delta \epsilon'$  plot shows similar minimum and maximum in the millimeter wave region. The average value of  $\Delta \epsilon'$  between ordinary and extra-ordinary ray appears to be 2.193 in this frequency range. This massive bire-fringent effect in crystalline sapphire is an advantage in prospective applications such as millimeter and submillimeter wave polarimeter, phase rotator, phase shifter, modulator and beam divider. The appearance of the maximum and minimum in the  $\Delta n$  and  $\Delta \epsilon'$  may lead to the use of sapphire as a switching device.

We have recently constructed a special dispersive Fourier transform spectrometer for direct experimental separation of complex magnetic permeability spectra from total or combined complex dielectric permittivity and complex magnetic permeability spectra of ferrite materials in the millimeter and submilli-



meter wavelength range. The induced ferromagnetic resonance moves to higher frequency as the applied external high intensity magnetic field is increased to higher values. As the frequency as well as the external applied high intensity magnetic field can be varied continuously, one can easily observe the quencing effect and resonance line widths. A natural ferromagnetic resonance has been observed in a radiation absorbing material. The center of the natural resonance line lies around 250 GHz.

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